DEVELOPMENT OF A STANDARD REFERENCE MATERIAL FOR THE FLUID POWER INDUSTRY: ISO MEDIUM DUST IN OIL

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ABSTRACT

The T2.9 Contamination Technology Committee of the National Fluid Power Association has requested the development of a Standard Reference Material (SRM 2806) consisting of an ISO Medium Test Dust suspended in MIL-H-5606 hydraulic fluid. SRM 2806 is being certified for particle size distribution, to be used in conjunction with the NFPA "Hydraulic Fluid Power - NIST Traceable Calibration Method for Liquid Automatic Particle Counters". Two associated uncertified reference materials composed of dry ISO Medium Test Dust (RM 8631) and ISO Ultra Fine Test Dust (RM 8632) will be available.

The size distribution and concentration of the particles in the SRM are being determined by electron microscopy coupled with image processing. Scanning electron microscopy (SEM) in the backscatter electron image mode provides a traceable method of size measurement with high contrast and imaging capabilities for small particles. Image processing software, developed at NIST, uses thresholding and "blobbing" to derive the particle size distribution from archived images, images that will be available for future reference and analysis. We have made comparisons between the particle size distributions obtained from an optical particle counter using an extinction sensor and from image processing.

INTRODUCTION

The national fluid power industry utilizes optical particle counter (OPC) technologies to assess the level of hydraulic oil contamination by suspended particulate which is often related to the integrity of the system and the usage of the fluid. OPC's are also used in various filter testing operations by the manufacturers and the users. ISO standard method 4402 has been used for 20 to 30 years to calibrate optical particle counters in terms of particle size as a function of particle concentration (1). The calibration material used in this standard method is Air Cleaner Fine Test Dust (ACFTD) produced in the past by a division of General Motors Corporation. This material consists of a polydisperse mineral dust, with the largest number of particles falling into the

size range of 1-20 μm diameter with a small fraction extending out to approximately 80 μm. Some problems have arisen with the employment of ACFTD in such calibration procedures. First, there has been ongoing concern that the particle size distribution is not accurate in the small particle (< 10 μm) size regime of the distribution (2,3,4,5). These researches and others had noted that there are more sub-10 μm particles in ACFTD than reported by ISO 4402. Second, and not least important, the production of ACFTD was discontinued by the supplier. Thus there is a need, recognized by the National Fluid Power Association (NFPA) Contamination Control Committee T 2.9, to design, investigate, and devise a new standard method (Hydraulic Fluid Power - NIST Traceable Calibration Method for Liquid Automatic Particle Counters) using a new Standard Reference Material (6). The T 2.9 Committee saw a need to have a material issued by a recognized certifying organization and thus requested the National Institute of Standards and Technology to develop a Standard Reference Material (SRM) for use by the fluid power industry. The new Standard Reference Material, currently under certification (designated by NIST as SRM 2806) is composed of ISO Medium Dust suspended in MIL-H 5606 hydraulic fluid. The particle size distribution and concentration will be certified for this material. There are associated reference materials (RM's) composed of dry ISO Medium and ISO Ultra Fine Test Dusts.

This paper presents the work in progress toward the certification of SRM 2806 and associated RM's. The particle size distribution is determined by electron microscopy utilizing image processing techniques. Our approach entails four major objectives: (1) material homogeneity testing, (2) particle filtration, (3) electron microscopy to obtain digital images of the separated material and (4) image analysis to provide the size distribution. The RM's, which will not be certified, have been spin riffled, packaged, and are undergoing homogeneity testing.

EXPERIMENTAL SECTION

Production of SRM 2806

The SRM material is produced by Fluid Technologies Inc.* (FTI) from ISO Medium Dust obtained from Powder Technologies Inc. (PTI) and tested for certification by NIST. The ISO Medium Dust, lot number 4390C, specified by ISO/WD 12 103 (7) is suspended at a concentration of 2.8 mg of dust per liter of MIL-H 5606 hydraulic fluid. 50 ppm of an antistatic agent is added to increase the electrical conductivity of the oil. The material is produced in 320-bottle batches, each bottle containing 400 mL of the suspension. Quality assurance for both production and testing was developed by a NFPA task force composed of members from FTI, HIAC/ROYCO, Nelson Industries, NIST and Pall Filter Corporation. Experiments were conducted by FTI and particle measurements were made by both FTI and NIST with NIST contributing to data analysis.

Homogeneity Testing/Batch Screening

Selected bottles from each batch are tested for homogeneity at both FTI and NIST using OPC's with extinction sensors calibrated to ISO 4402 (ACFTD standard) and monodisperse polystyrene latex spheres. A batch of bottles is deemed homogeneous if the size distributions determined for the sampled bottles have a coefficient of variation less than or equal to 4% for particle size < 10 μm, less than or equal to 7% at >30 μm and there are no systematic variations in the batch. The cumulative particle size distribution is determined for the nominal size range of 1-80 μm particle diameter and FTI and NIST measurements are compared for the same batch of materials. A sampling procedure was developed at NIST to measure the bottle-to-bottle homogeneity, but at the same time decouple any systematic error in the measurements due to possible instrument (in the OPC) drift. Four bottles (a, b,c,d) were sampled and analyzed from approximately the following four points in the production cycle: 5%, 30%, 60%, and 95% point. Then another set of four bottles that were produced directly adjacent to the first 4 were analyzed. For example, first 16 bottles 5a, 5b, 5c, 5d, 25(a,b,c,d), 50(a,b,c,d) and 75(a,b,c,d) were analyzed in that order. Then bottles 6(a,b,c,d), 26(a,b,c,d), 51(a,b,c,d), and 76(a,b,c,d) were analyzed all by the same calibrated OPC. With three replicates for each bottle, this totals to 96 measurements. Each batch of 320 bottles was subjected to this procedure or a modified version of this test.

Quantitative Particle Separation by Filtration

Particles are separated from the hydraulic fluid by filtration. All of the apparatus associated with the procedure is carefully cleaned with filtered solvents. The filtrations are performed in a class 100 clean room to avoid possible contamination by ambient airborne particles. Polycarbonate filters (25 mm diameter with 0.2 μm pore size) are used to filter the particles from the oil. These filters have high collection efficiency for particles > 0.2 μm and provide a smooth, planar surface for electron microscopy (8,9,10). Filtered heptane was used as the clean solvent to remove the hydraulic oil from the filter and to wash the filtering apparatus.

The procedure, adapted from an existing SAE Method (11) entails three steps per sample producing 3 filters for analysis: (1) the filter, solvent and apparatus blank is obtained by flushing several hundred milliliters of filtered solvent through a clean filter while at the same time washing the walls of the filter funnel, (2) filtering a known volume (at STP) of SRM 2806 hydraulic oil suspension through a second filter followed by funnel wall washing and (3) extensively washing the funnel down with filtered solvent onto a third clean filter. The first procedure provides assessment with respect to the cleanliness of the blank filter material, of the filtering apparatus, of the filtered solvent, and of the overall sample processing. Then in the second procedure individual bottles of

SRM 2806 are sonicated, mechanically shaken, and then resonicated. Following resuspension and mixing, small, known volumes are carefully pipetted from the bottle and flushed through the filter using the prefiltered solvent. The walls of the funnel and pipette are extensively flushed with solvent. Finally, for the third procedure a new clean filter is installed and the same funnel is washed down again to assure that all particles have been removed from the walls. All three filters are examined by electron microscopy and the measurement results obtained from them are used in the data analysis.

Electron Microscopy

Electron microscopy is used in this work as the primary particle size/count measurement methodology. Scanning electron microscopy (SEM) in the backscatter electron imaging mode is used because it spans the range of particle size of concern, 0.5 μm to 80 μm, and provides the maximum grey level contrast for subsequent processing of the digital images. The technique is well established (10,12,13,14,15). The entire 25 mm diameter polycarbonate filter containing the particle sample is gold-coated using a low temperature plasma source and subsequently mounted onto the sample stage in the SEM. The SEM is automated with computer control for the sample stage movement and data collection. The computer selects a true set of random fields-of-view to be collected and computes the most efficient route to scan across the sample surface. The stage then steps through the sequence, the sample is brought to optical focus, and backscatter electron images are collected and stored digitally for each field. This is illustrated in Figure 1 where a random field selection from a circular filter surface is simulated and one of the associated electron images is "grabbed" by the interfaced computer. All images are archived on two CD-ROMS so that a permanent record of the data will be available. Magnifications of 100X, 500X and 3300X are used to interrogate and span the particle size range of interest. These magnifications correspond to field-of-view with areas of approximately 0.8 mm^2, 0.03 mm^2, and 0.0008 mm^2. Figure 2 presents three typical micrograph images of the ISO Medium Dust on a polycarbonate filter at the three magnifications 2a - 3300X, 2b - 500X, and 2c -100X. These micrographs show the number of particles and relative size and shape of the dust material. SRM 484f, a scanning electron magnification standard, is mounted in the x and y direction (orthogonal) on the SEM sample stage and used in conjunction with each sample to calibrate the x-y length for particle sizing. SRM 1690, 1 μm polystyrene spheres, was examined by the same procedures used for the dust particles to verify the procedure. Elemental analysis is conducted for a subset of dust particles in the filter sample using energy dispersive x-ray spectroscopy to assure that only mineral dust is being analyzed and that other contaminating particulate material is not present.

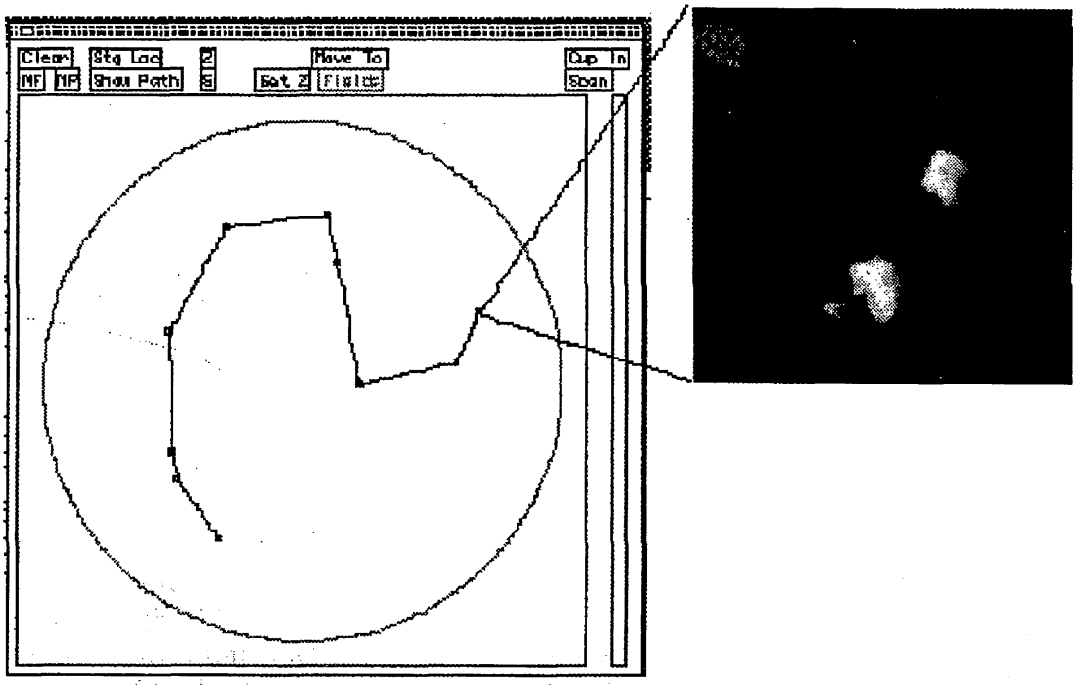

Figure 1. Screen display on the Macintosh computer used to randomly select fields-of-view and collect micrograph images. Shown here is a 3300X image of ISO Medium Dust. The analysis path is shown and is independent of the random field selection.

Image Analysis for Particle Size and Number Determination

The SEM micrograph images are composed of digitized 8-bit grey level points commonly referred to as pixels. The size scale for each pixel is determined by the magnification of the SEM which is accurately measured using the magnification standard, SRM 484f. The dust, being composed of mineral particles, has high image contrast due both to the particle topography and to the fact that the average elemental atomic number is larger than the substrate filter material that is composed of carbon and hydrogen. The particles appear as "light" objects because of an increase electron backscatter yield over the filter. The pixels that define the particles' size and shape are identified by a well known method of grey-level thresholding that sets the lower grey level that a mineral particle can have. Thresholding is done for each image, and normally a minimum pixel area object limit is set. For example, one setting would allow particles to be counted if they are composed of more than 5 pixels. After the image is thresholded, the total pixel area for each particle is computed as well as the longest chord length (maximum particle diameter), and particle perimeter. By summing the number of thresholded objects, the particle number for any given field-of-view at whatever magnification is determined from the image. An

example of a SEM raw image (A) and thresholded regions (B) are shown in Figure 3. In Figure 3C, certain particles are accepted for the data set because their total pixel area exceeds a discrimination level set by the user.

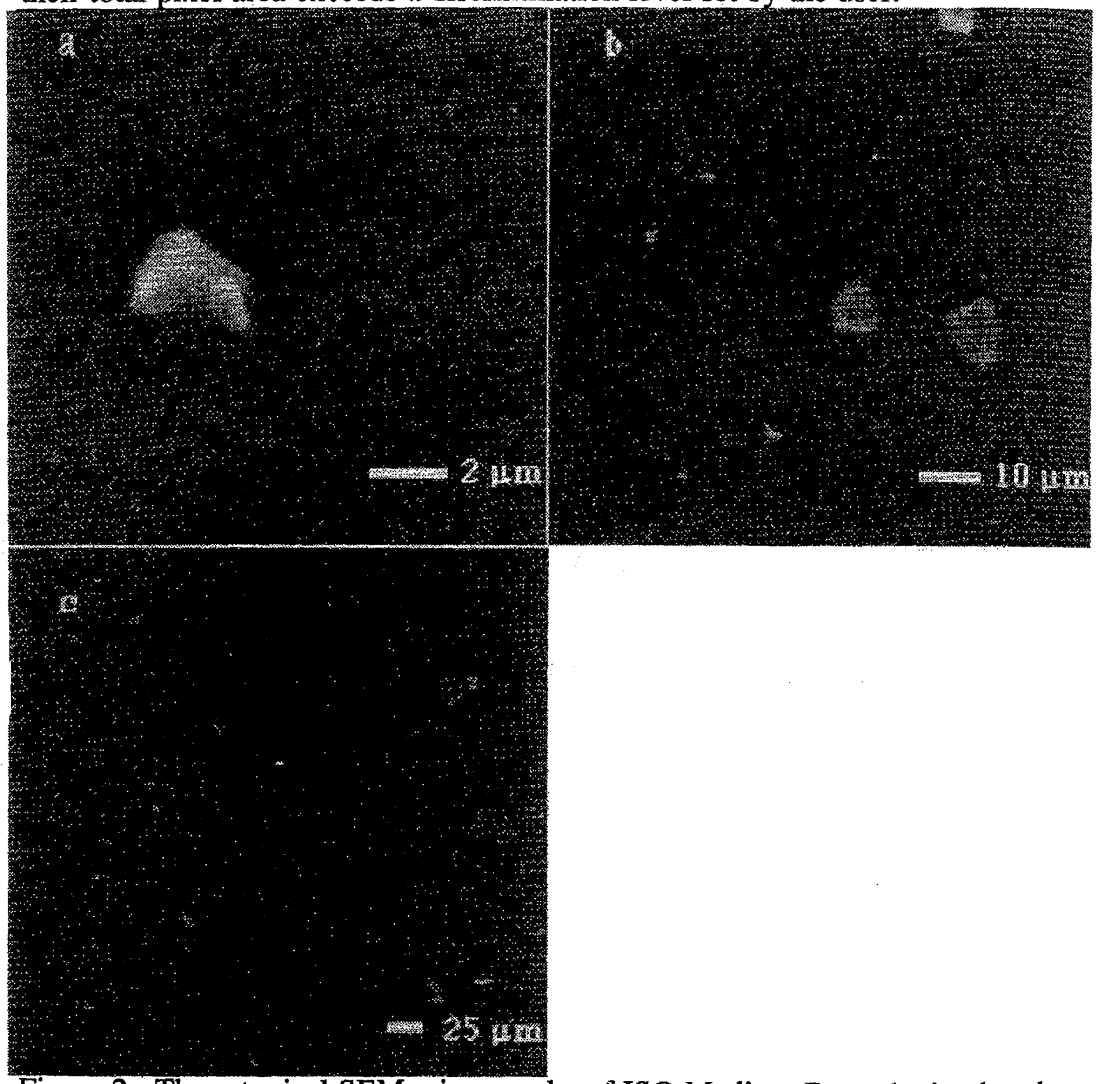

Figure 2. Three typical SEM micrographs of ISO Medium Dust obtained at the three magnifications.

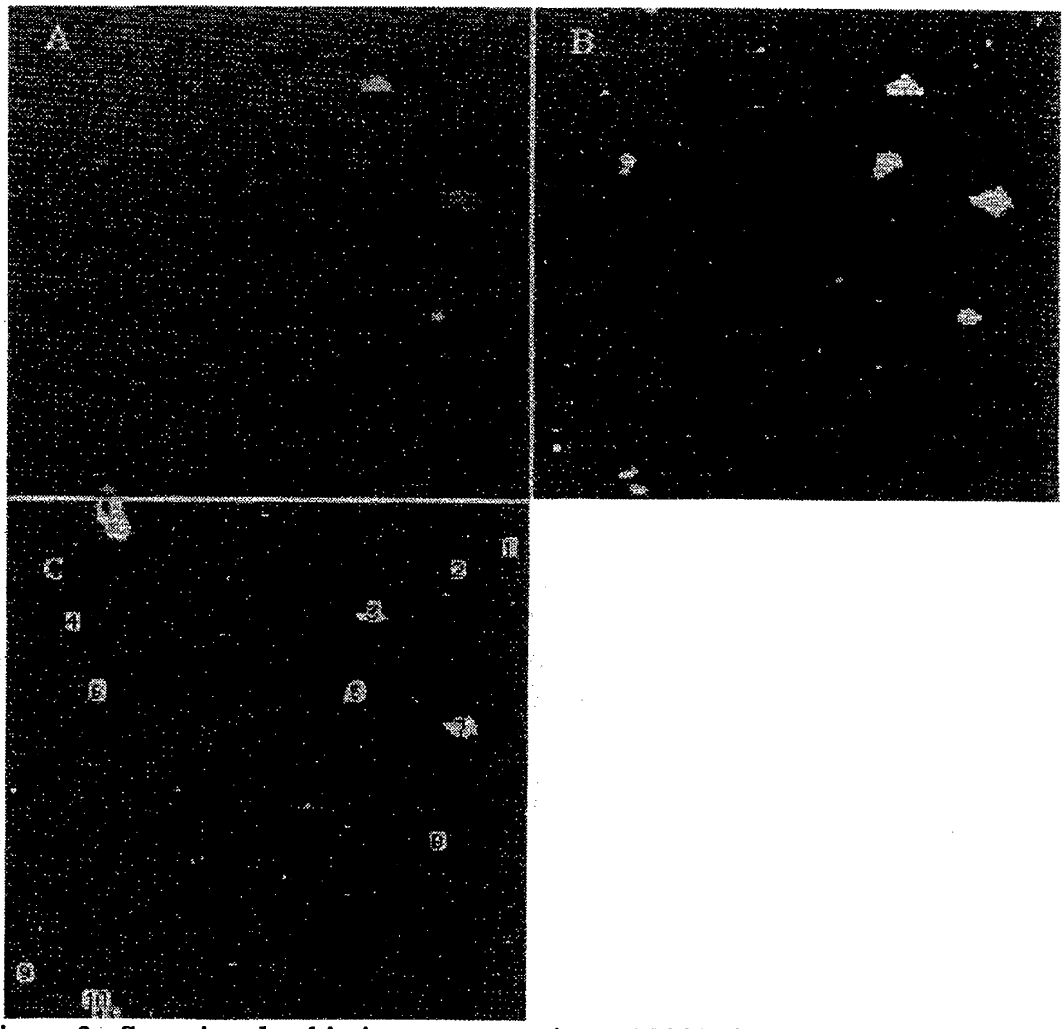

Figure 3. Steps involved in image processing a 3300X SEM image of ISO Medium Dust. (A) is the original image, (B) is the grey-level thresholded image, and (C) shows particles collected and rejected. The small were not accepted because of low pixel count discrimination and one large particle was rejected because it touches the image boundary.

Public domain computer software, MacLispix, developed at NIST and NIH Image is used to do the image processing (16,17). The relevant information derived from the images is the following: particle area, longest diameter, particle perimeter, number of detected objects, location of the field-of-view on the filter and location of each particle thresholded. Since the micrograph images are carefully archived, additional analyses can be done any time in the future.

Stability Testing Of SRM 2806

SRM 2806 will be tested at 6 month intervals to assure that the particle size distribution is not changing with time. Optical particle counters calibrated to standard polystyrene spheres will be used for this stability monitoring. A historical record of the size distribution will be made from the time a batch of material arrives at NIST until it is sold. Spot microscopy checks will be performed on selected bottles as necessary if the OPC measurements indicate any changes in the material.

Reference Materials 8631 and 8632

Reference materials 8631 and 8632 are composed of ISO Medium and ISO Ultra Fine Dry Dust from PTI lot numbers 4390C (same lot as the SRM 2806) and 4476J. The material was received from PTI in 3.6 kg bottles. This dust was dried and spin-riffled into 20 g aliquots. The spin-riffler could accommodate 147 small sample bottles at once. The material is being examined for homogeneity using optical particle counters and by x-ray diffraction.

RESULTS AND DISCUSSION

Our results, at this time, must be qualified since we are reporting preliminary data and not the certified values. Homogeneity testing gives us a measure of the within batch variability for the SRM and a measure of the batch-to-batch uniformity. The relative standard deviation for within batch measurements is less than 2% for the particle size fraction <10 μm. Figure 4 shows the batch-to-batch comparison in histogram form. The histogram is composed of the mean values of the cumulative particle counts for the same volume of fluid analyzed. The differences among batches are on the order of 1% to 6% across the entire size distribution.

We have observed non-uniform particle deposition on many of the filters after dust separation from the hydraulic oil. Figure 5 shows a schematic of a filter with the regions from which micrograph field-of-view images were sampled and their respective particle counts represented in grey-level format.

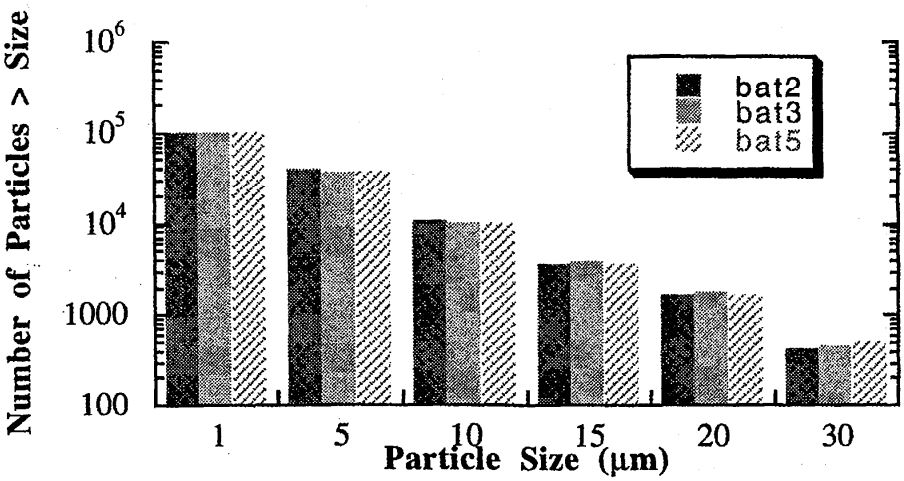

Figure 4. Histogram representation summarizing the results of homogeneity testing 3 batches of ISO Medium Dust in hydraulic fluid. The mean values of the cumulative particle counts are represented along the ordinate for each batch. The absissa corresponds to OPC-diameter from the ISO 4402 calibration.

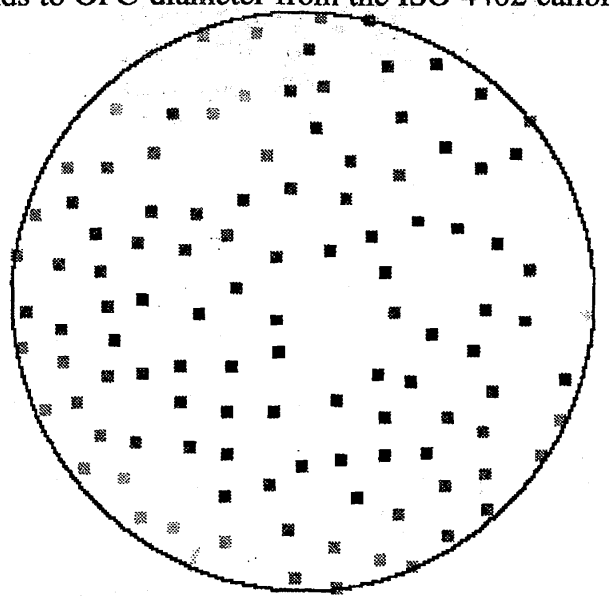

Figure 5. Schematic of a filter surface showing the location from which fields were sampled and micrographs obtained. The dark squares correspond to fields with the highest number of particles counted.

The darkest squares contain the largest count and are generally located in the interior area of the filter surface, while the low-count fields are found closer to the edge of the surface. Note that there are fields-of-view that overlap the boundary as required for correct sampling. If the boundary is a non-allowed sampling area, sampling would not be random, but biased. These edge regions are corrected by scaling the particle-free area observed in the micrographs. To get a representative particle count we must randomly sample the filter surface during the image collection process. A statistical analysis method used in the mining industry is being applied to derive the number of particles collected on the filter from the discrete fields-of-view data.

Figure 6 presents the results of measurements on the new SRM 2806 material using an OPC with an extinction sensor calibrated to ACFTD-ISO 4402. This figure shows the calculated cumulative size distribution for 1 mL of a 1 mg/L dust-in-oil suspension (1 µg of dust). The mean values for 96 measurements on the SRM 2806 are presented. It can be seen that although the size distributions are similar in this measurement, SRM 2806 has relatively more small particles and fewer large particles than the ACFTD ISO 4402 material. The two distributions intersect at approximately 13 µm. The standard error of the mean for 96 measurements ranges from 0.06% at > 1 µm to 0.6% at > 30 µm.

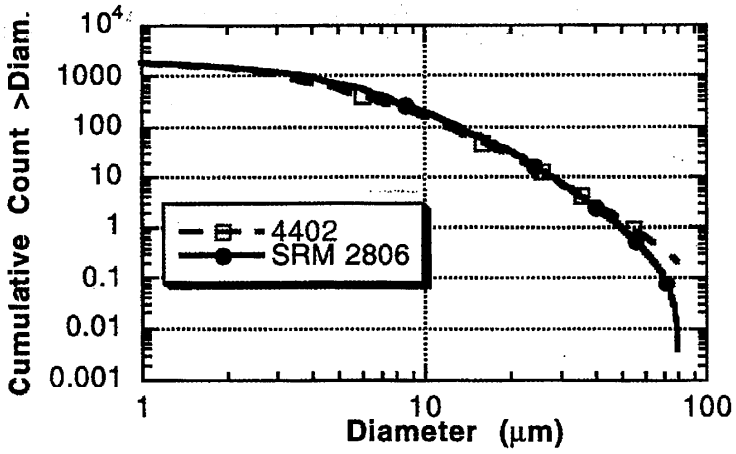

Figure 6. Plot of measurement results showing cumulative particle size distributions for ISO Medium Dust and ACFTD both obtained from an OPC calibrated to ISO 4402 and referenced to 1 µg of dust.

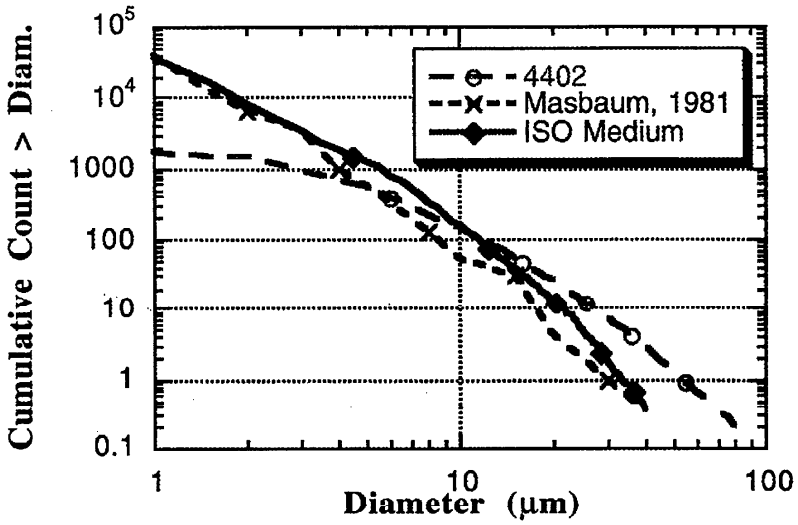

Figure 7. Plot of ISO 4402 values (1), Masbaum's (3) ACFTD measurements made by microscopy, and ISO Medium Dust measurements by SEM/image processing at NIST. All measurements referenced to 1 μg of material.

The measurements made using an optical particle counter calibrated to ISO 4402 specifications are compared to actual particle counting by microscopy/image processing. These preliminary results found in Figure 7 show the ISO 4402 curve , the values obtained by Masbaum who used wet sieving and microscopy to measure the size distribution of ACFTD, and the microscopy results for ISO Medium Dust extracted from the SRM 2806. Masbaum and SRM data are expressed as equivalent spherical diameters while the ISO 4402 is longest chord. Our work on the ISO Medium Dust indicates that the size distribution is similar to that found by Masbaum on ACFTD. Both Masbaum and NIST data indicate the presence of more small (<10 μm) particles than identified by ISO 4402 as reported by others (2,3,4).

We are in the process of assessing the uncertainty of the measurements. There are numerous possible sources of random and systematic error. Although not intended as an exhaustive error analysis, an outline of some of the possible error sources are presented. A systematic error likely to be present in the data is due to the fact that the microscopic images are collected on particles laying flat on a planar surface and thus exhibiting their largest projected area whereas in the OPC application, the material is normally employed with the particles suspended in random orientation or oriented with respect to the fluid flow. Figure 8 plots the tabulated data of Ellison (18) who characterized silica particles by light microscopy cataloging equivalent circular areas for particle populations ranging

in size from 150 to 660 particles. Plotted are the mean areas for the mean particle size. To obtain random orientation, the author suspended similar silica particles in agar. The particles in a planar orientation have, as expected, an area ranging from 11% to 33% larger than the agar-mounted silica material.

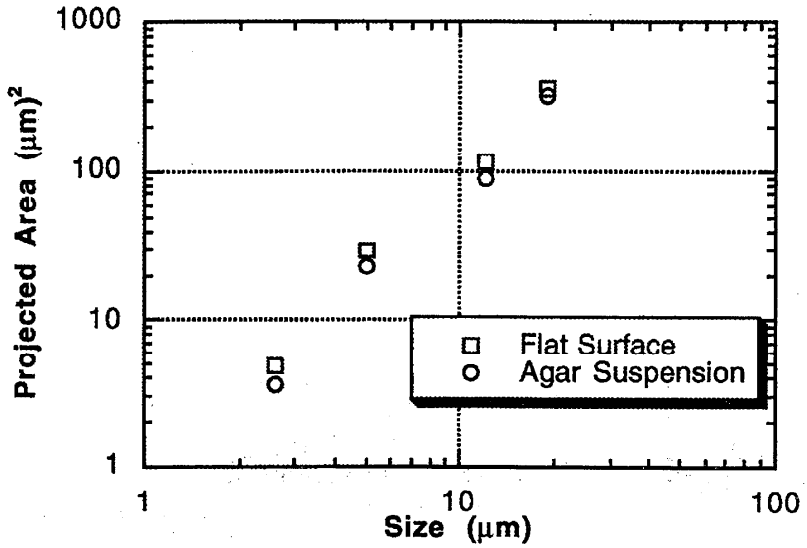

Figure 8. Plot of mean particle size versus average projected area for silica particles laying flat on a microscope stage and suspended in agar with random orientation. Data taken from a table presented by Ellison (18).

Errors can be present in oil filtering from volume uncertainties, particle loss in the filtering process (adhering to funnel walls), particle agglomeration and non-uniform filter coverage through the sampling process. From the microscopy, there are errors in determining the magnification. In image processing there is quantization error associated with the digitization/pixel representation of the micrograph image and thresholding uncertainty.

In Summary

In summary, this paper reports work in progress on a new SRM 2806 ISO Medium Test Dust in oil material for the fluid power industry. Work on this standard was initiated by the NFPA Committee T2.9 because of a real need in the fluid power community. This paper presents the experimental design and early results as well as discussing some of the pitfalls of the measurement process. Efforts are being made to have this SRM available to the user in 1996.

ACKNOWLEDGMENTS

The authors would like to acknowledge valuable contributions from members of FTI, HIAC/ROYCO, Nelson Industries, and Pall Corporation.

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* Commercial equipment, instruments, materials, or software are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommmendation or endorcement of these items by the NIST, nor does it imply that they are the best available for the purpose.

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